

TRANSMITTAL LETTER TO THE UNITED STATES

JMYT-253US

DESIGNATED/ELECTED OFFICE (DO/EO/US)

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR

CONCERNING A FILING UNDER 35 U.S.C. 371

10/019903

INTERNATIONAL APPLICATION NO.

PCT/GB00/02387

INTERNATIONAL FILING DATE

20 June 2000 (20.06.00)

PRIORITY DATE CLAIMED

24 June 1999 (24.06.99)

TITLE OF INVENTION

PROCESS FOR THE REGENERATION OF REFORMING CATALYSTS

APPLICANT(S) FOR DO/EO/US

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Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371 (c) (2))
 - a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ A copy of the International Search Report (PCT/ISA/210).
8. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
9. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
10. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
11. ☒ A copy of the International Preliminary Examination Report (PCT/IPEA/409).
12. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).

Items 13 to 20 below concern document(s) or information included:

13. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
14. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
15. ☐ A **FIRST** preliminary amendment.
16. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
17. ☐ A substitute specification.
18. ☐ A change of power of attorney and/or address letter.
19. ☒ Certificate of Mailing by Express Mail
20. ☐ Other items or information:

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21 DEC 2001

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21. The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)) :

- ☐ Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO \$970.00
- ☒ International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO \$840.00
- ☐ International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$690.00
- ☐ International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$670.00
- ☐ International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4) \$96.00

ENTER APPROPRIATE BASIC FEE AMOUNT =**\$890.00**

Surcharge of \$130.00 for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492 (e)).

\$0.00

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE
Total claims	16 - 20 =	0	x \$18.00
Independent claims	2 - 3 =	0	x \$84.00

\$0.00**\$0.00**

Multiple Dependent Claims (check if applicable).

☒**\$280.00****TOTAL OF ABOVE CALCULATIONS =****\$1,170.00**

Reduction of 1/2 for filing by small entity, if applicable. Verified Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28) (check if applicable).

☐**\$0.00****SUBTOTAL =****\$1,170.00**

Processing fee of \$130.00 for furnishing the English translation later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492 (f)).

+

\$0.00**TOTAL NATIONAL FEE =****\$1,170.00**

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) (check if applicable).

☐**\$0.00****TOTAL FEES ENCLOSED =****\$1,170.00**

Amount to be:
refunded \$
charged \$

- ☒ A check in the amount of **\$890.00** to cover the above fees is enclosed.
- ☐ Please charge my Deposit Account No. _____ in the amount of _____ to cover the above fees.
A duplicate copy of this sheet is enclosed.
- ☒ The Commissioner is hereby authorized to charge any fees which may be required, or credit any overpayment to Deposit Account No. **18-0350** A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

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REGISTRATION NUMBER

December 21, 2001

DATE

PROCESS FOR THE REGENERATION OF REFORMING CATALYSTS

The present invention relates to methods for the regeneration of catalytic reactors.

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Catalytic converters are frequently used in internal combustion engines in order to meet the various regulations concerning the levels of pollutants in exhaust gases. A three-way catalyst is a common form of converter used. This has three main duties, namely, the oxidation of CO, the oxidation of unburnt hydrocarbons (HC's) and the
10 reduction of NOx to N2. Such catalysts require careful engine management to ensure that the engine operates at or close to stoichiometric conditions, that is fuel/air $\lambda=1$. Growing awareness of the need to conserve the earth's resources and increasingly stringent legislation have recently prompted a search for cleaner and more efficient alternatives to the internal combustion engine. One of the most promising of these is the
15 combination of an electric motor and a fuel cell. However, the latter requires a source of hydrogen, for which there is no supply and distribution infrastructure comparable to that for liquid fuels.

The use of fuel cells is not just limited to vehicle applications. Emergent markets
20 include domestic co-generation of heat and power, and power generation in remote locations. Again, the availability of hydrogen is a key issue, with natural gas often being the preferred fuel for domestic systems, and liquid hydrocarbons being more transportable to remote locations.

25 The problems of hydrogen supply and distribution can be overcome by generating it within the fuel-cell system. A hydrogen-rich gas stream, commonly known as 'reformat', can be produced by catalytically converting organic fuels (such as gasoline, natural gas or alcohol). The process, which is referred to as 'reforming' or 'fuel-processing', can occur by a number of different reaction mechanisms :

- 30
- dissociation (splitting of the fuel molecules),
 - steam reforming (reaction of the fuel with H₂O),
 - partial oxidation (reaction of the fuel with O₂, usually supplied as air)
 - combinations of the above reactions.

Because the performance of the reformer has a direct effect on the power output from the fuel cell, deactivation of the catalyst cannot be allowed to proceed unchecked. However the process of power generation cannot be stopped to allow regeneration of the reformer, when its performance falls below an acceptable level. The regeneration of such catalytic reactors whilst they are in use is the problem this invention sets out to solve.

In many instances, the performance of the catalyst declines while it is being used. Although the rate of decline can usually be reduced by changing the operating conditions, it is often accompanied by a drop in hydrogen and power output. One frequently practised method for restoring the performance of a fuel processor is to replace the catalyst periodically. Another method involves regenerating the catalyst while it remains in the processor, using a prescribed regeneration procedure (eg see JR Rostrup-Nielsen in Catalysis Today, Vol 37, 1997, p 225-232). However, both these require that the process of hydrogen-generation is interrupted in order to restore performance.

Thus in a first aspect, the present invention provides a method for regenerating a catalytic fuel processor, while it is being used to supply hydrogen to a fuel cell, comprising any one or more of the steps of:

- continuing to pass fuel, air and steam through a reforming catalyst whilst the catalyst is heated by an external heat source such that the temperature of the catalyst may be adjusted,
- continuing to pass fuel, air and steam through a reforming catalyst and modulating the air and/or steam feed rate,
- continuing to pass, air, fuel and steam through a reforming catalyst and modulating the feed-rate of the fuel.
- continuing to pass fuel, air and steam through a reforming catalyst wherein an oxygenate additive is added to the feed.

and maintaining the hydrogen concentration in dry reformat above 25% throughout the operation of the processor.

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The reforming catalyst may be one component of the catalytic fuel processor, or it may be the only component. Where loss of performance is being caused by a particular component in a complex fuel mixture, the regeneration method can be targeted at the specific deactivating effect.

Thus in a second aspect, the present invention provides a method for preventing or retarding the de-activation of a catalytic fuel processor while it is being used to supply hydrogen to a fuel cell comprising any one or more of the steps of:

- continuing to pass fuel, air and steam through a reforming catalyst whilst the catalyst is heated by an external heat source such that the temperature of the catalyst may be adjusted,
- continuing to pass fuel, air and steam through a reforming catalyst and modulating the air and/or steam feed rate,
- continuing to pass, air, fuel and steam through a reforming catalyst and modulating the feed-rate of the fuel,
- continuing to pass fuel, air and steam through a reforming catalyst wherein an oxygenate is added to the feed.

and maintaining the hydrogen concentration in dry reformat above 25% throughout the operation of the processor.

Again, the reforming catalyst may be one component or the only component of the catalytic fuel processor. In a preferred embodiment of the invention water is temporarily added to the feed. ('Feed' is a term used to describe the reactant mixture supplied to the fuel processor.) The water will be rapidly converted into steam in the catalyst. The water (steam) to carbon ratio in the feed may readily be adjusted in order to permit targeting of the regeneration method to a specific deactivating event. Adjusted for the purposes of this invention means increased or decreased. The oxygen to carbon ratio may also be adjusted for the same purpose, by adding or changing the feed-rate of air, or by changing the feed-rate of the fuel.

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The catalyst may contain one or more base metals, which may include copper (often used for reforming methanol) and nickel (used for reforming natural gas and higher hydrocarbons). Alternatively, the catalyst may contain one or more precious metals, which may include gold, platinum, palladium, iridium, silver, rhodium and ruthenium. Furthermore, it may contain both base metal(s) and precious metal(s). The catalyst may also contain refractory materials, such as ceramics, metal oxides, perovskites, metal carbides and metal sulphides.

Additives may be added to the feed for various purposes, including acceleration of start-up of a fuel-processor, and the prevention or inhibition of its deactivation. Alternatively, a fuel to which additives have been added during manufacture can be used. In a preferred embodiment of the invention, the additive is an oxygenate, and in an especially preferred embodiment the oxygenate is MTBE (methyl-tert-butylether).

In order to facilitate the regeneration procedure, the temperature of the catalyst bed may be raised temporarily by an external energy source. Within a fuel cell system, the external energy source may be an electrical heater, or a burner (which combusts either some of the fuel or some of the hydrogen produced). In addition, or alternatively, the temperature of one or more of the feed components may be raised temporarily, again by an external heat source.

Among the most common causes of deactivation of a fuel-processor is the retention of carbon or sulphur species by the catalyst. Therefore, in a further embodiment of the invention, catalyst regeneration may occur by removal of the carbon or sulphur species. The carbon species originate from the fuel molecules, and indicate the occurrence of undesired side reactions. The sulphur species originate either from indigenous contaminants in the fuel or from compounds deliberately added to the fuel (such as the odourants used to give natural gas its recognisable smell).

The present invention will now be described by way of the following examples in which:

Figure 1a: shows the composition of dry reformat as a function of time during reforming of dodecane (under conditions described in Example 1).

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Figure 1b shows the effect of air pulses on the composition of dry reformat during reforming of dodecane (as described in Example 2).

In both cases % in reformat is on the vertical axis and time/hours is on the horizontal axis.

5 ♦ represents Hydrogen, ■ represents carbon dioxide, and ▲ represents carbon monoxide.

Figure 2a shows the composition of dry reformat as a function of time during the reforming of toluene under the two conditions described in example 5.

10 Figure 2b shows the effect of increasing the air feed-rate on the composition of dry reformat, during reforming of toluene (as described in example 6).

In both cases the left hand side of the vertical axis represents % in reformat and the right hand side of the vertical axis represents temperature in °C. The horizontal axis represents time/hours; ♦ represents hydrogen, ■ represents carbon dioxide, ▲ represents carbon monoxide and x represents temperature.

15 It will be appreciated that many variations can be made to the invention herein described without departing from the present inventive concept.

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Example 1 (Aliphatic fuel ; no regeneration)

A bed (0.2 g) of reforming catalyst was packed into a tubular quartz reactor, which was positioned at the centre of a furnace. A mixture of dodecane vapour (produced by vaporising the liquid at a rate of $4 \text{ cm}^3 \text{ hour}^{-1}$), air ($200 \text{ cm}^3 \text{ min}^{-1}$) and
25 steam (produced by vaporising water at a rate of $4 \text{ cm}^3 \text{ hour}^{-1}$) was passed through the catalyst bed, which was heated by the furnace. The furnace temperature was maintained at 500°C . The product stream (ie the reformat) passed through a drier before entering a gas chromatograph, which was used to analyse for hydrogen. The initial concentration of hydrogen in the dry reformat was just above 25%, but declined to 20% within an
30 hour, and to 15% within 4 hours. See Figure 1a for results.

Example 2 (Regeneration by air pulses)

The test procedure described in Example 1 was repeated with a fresh charge of catalyst, except that every 10 minutes the air feed-rate was increased to $350 \text{ cm}^3 \text{ min}^{-1}$ for 30 seconds. Apart from the duration of the extra air pulses, the concentration of hydrogen in the dry reformat remained above 25% during 3 hours of testing. See Figure 1b for results.

Example 3 (Regeneration by temperature excursions)

The test procedure described in Example 1 was repeated with a fresh charge of catalyst. Whenever the hydrogen concentration in the dry reformat dropped below 25%, it could be restored by raising the furnace temperature to 600°C for 1 minute.

Example 4 (Inhibition of de-activation by MTBE addition)

The test procedure described in Example 1 was repeated with a fresh charge of catalyst, except that 10% (by volume) methyl-tert-butylether was added to the dodecane. The hydrogen concentration in the dry reformat remained above 25% throughout 5 hours of testing.

Example 5 (Aromatic fuel ; no regeneration)

A bed (0.2 g) of reforming catalyst was packed into a tubular quartz reactor, which was positioned at the centre of a furnace. A mixture of toluene vapour (produced by vaporising the liquid at a rate of $4 \text{ cm}^3 \text{ hour}^{-1}$), air ($175 \text{ cm}^3 \text{ min}^{-1}$) and steam (produced by vaporising water at a rate of $4 \text{ cm}^3 \text{ hour}^{-1}$) was passed through the catalyst bed, which was heated by the furnace. The furnace temperature was maintained at 500°C . The initial concentration of hydrogen in the dry reformat was 33%, but declined to 25% within 3 hours. When the catalyst was replaced by a fresh charge and the feed-rate of steam was doubled (by increasing the rate of vaporising water to 8 cm^3

hour⁻¹), the initial concentration of hydrogen in the dry reformat was 37%. Within 3 hours, the hydrogen concentration had declined to 30%. See Figure 2a for results.

Example 6 (Prevention of de-activation by extra air)

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The test procedure described in Example 5 was repeated with a fresh charge of catalyst, except that the air feed-rate was increased to 200 cm³ min⁻¹. The initial concentration of hydrogen in the dry reformat was 32%. The concentration remained unchanged during 3 hours of testing. See Figure 2b for results.

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CLAIMS

1. A method for regenerating a catalytic fuel processor, while it is being used to supply hydrogen to a fuel cell, comprising any one or more of the steps of:

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- continuing to pass fuel, air and steam through a reforming catalyst whilst the catalyst is heated by an external heat source such that the temperature of the catalyst may be adjusted,
- continuing to pass fuel, air and steam through a reforming catalyst and modulating the air and/or steam feed rate,
- continuing to pass, air, fuel and steam through a reforming catalyst and modulating the feed-rate of the fuel.
- continuing to pass fuel, air and steam through a reforming catalyst wherein an oxygenate is added to the feed.

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and maintaining the hydrogen concentration in dry reformat above 25% throughout the operation of the processor.

2. A method for preventing or retarding the de-activation of a catalytic fuel processor while it is being used to supply hydrogen to a fuel cell comprising any one or more of the steps of:

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- continuing to pass fuel, air and steam through a reforming catalyst whilst the catalyst is heated by an external heat source such that the temperature of the catalyst may be adjusted,
- continuing to pass fuel, air and steam through a reforming catalyst and modulating the air and/or steam feed rate,
- continuing to pass air, fuel and steam through a reforming catalyst and modulating the feed-rate of the fuel.
- continuing to pass fuel, air and steam through a reforming catalyst wherein an oxygenate is added to the feed.

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and maintaining the hydrogen concentration in dry reformat above 25% throughout the operation of the processor.

3. A method according to either claim 1 or claim 2, whereby water is temporarily added to the fuel.

4. A method according to claims 1 or 2 in which air is temporarily added to the feed.

5. A method according to claims 1 or 2 in which an oxygenate is added to the feed.

6. A method according to claim 5 in which the oxygenate is MTBE (methyl-tert-butylether).

7. A method according to claims 1 or 2 in which the catalyst bed temperature is raised temporarily by an external heat source.

8. A method according to claims 1 or 2 in which the temperature of one or more of the reactant feeds is raised temporarily.

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RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA,
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CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

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For two-letter codes and other abbreviations, refer to the "Guid-
ance Notes on Codes and Abbreviations" appearing at the begin-
ning of each regular issue of the PCT Gazette.

(54) Title: PROCESS FOR THE REGENERATION OF REFORMING CATALYSTS

(57) Abstract: The present invention relates to methods for the regeneration of catalytic reactors. In particular it relates to methods for regenerating a fuel-processing catalyst whilst it is still being used to supply hydrogen to a fuel cell. The temperature of the catalyst may be adjusted, the air, steam or fuel feed rate may be adjusted. Alternatively, additives may be added to the feed.

WO 01/00524 A1

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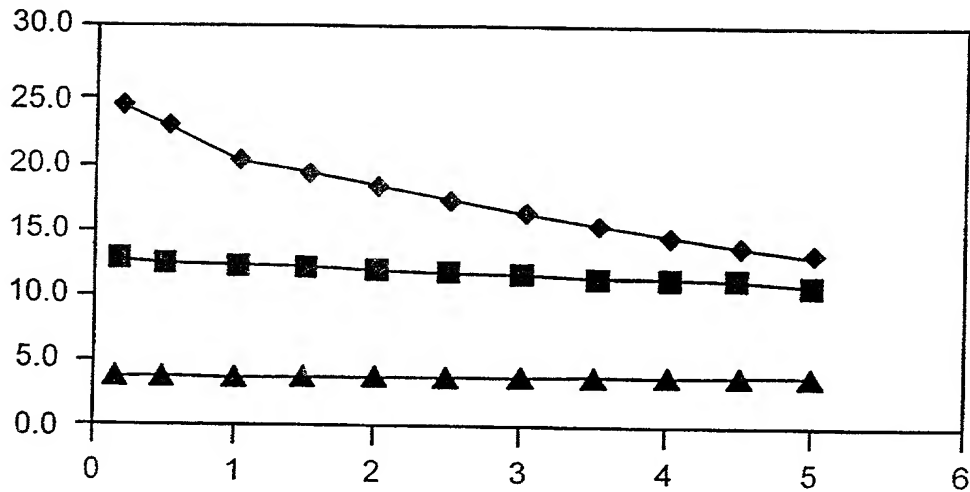


FIG. 1a

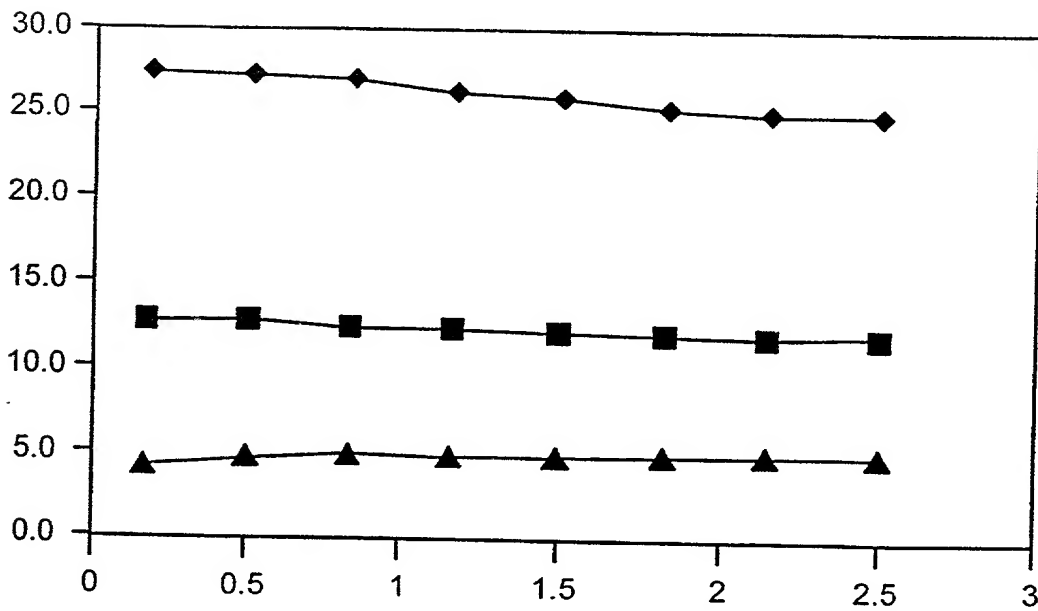
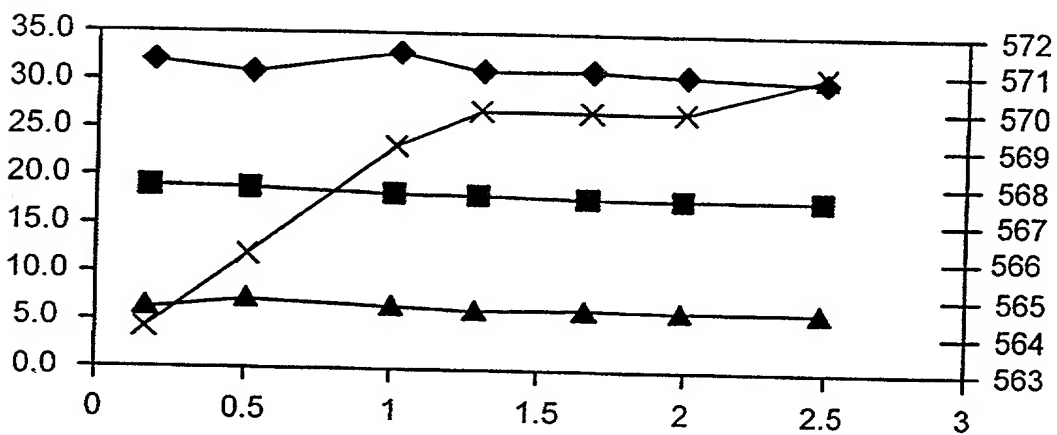
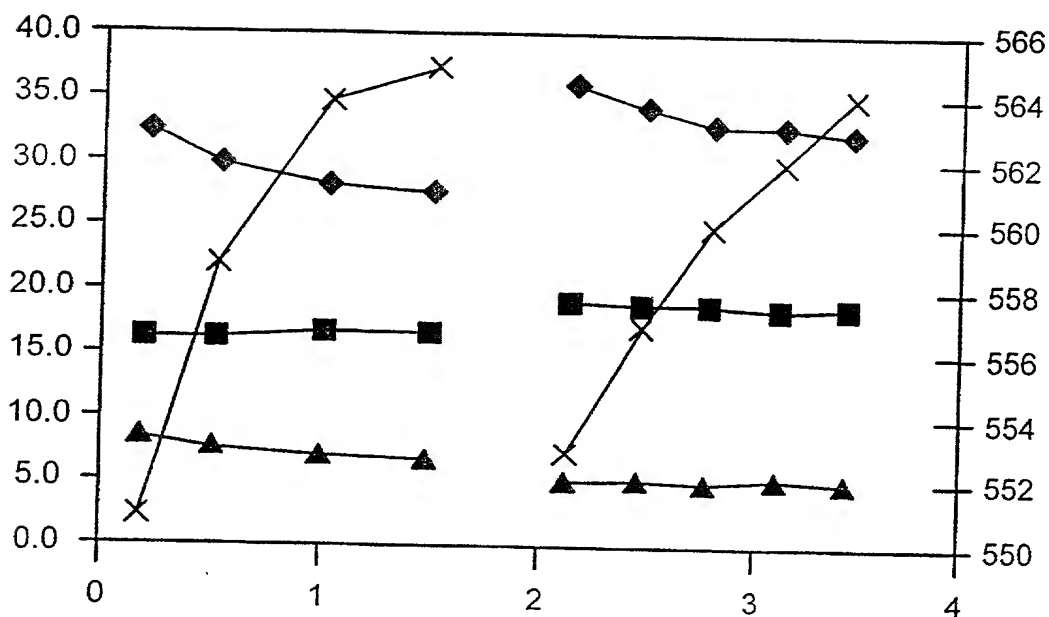


FIG. 1b

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Declaration and Power of Attorney For Patent Application

English Language Declaration

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

PROCESS FOR THE REGENERATION OF REFORMING CATALYSTS,

the specification of which is attached hereto unless the following box is checked:

☒ was filed on December 21, 2001 as
United States Application Number or PCT International Application Number 10/019,903
and was amended on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56.

I hereby claim foreign priority benefits under 35 U.S.C. § 119(a)-(d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s)

Priority Not Claimed

9914662.3

Great Britain

24 June 1999

(Number)

(Country)

(Day/Month/Year Filed)

☐

(Number)

(Country)

(Day/Month/Year Filed)

☐

I hereby claim the benefit under 35 U.S.C. § 119(e) of any United States provisional application(s) listed below.

(Application Number)

(Filing Date)

(Application Number)

(Filing Date)

I hereby claim the benefit under 35 U.S.C. § 120 of any United States application(s), or 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

PCT/GB00/0238720 June 2000Abandoned

(Application Number)

(Filing Date)

(Status - patented, pending, abandoned)

(Application Number)

(Filing Date)

(Status - patented, pending, abandoned)

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith:

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Paul F. Prestia	Reg. No. <u>23,031</u>	Allan Ratner	Reg. No. <u>19,717</u>	James C. Simmons	Reg. No. <u>24,842</u>
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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of sole or first inventor (given name, family name) Robert Burch

Inventor's signature R Burch

Date 15/04/2002

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Second Inventor's signature S Golunski

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☒ Additional inventors are being named on separately numbered sheets attached hereto.

3-00
Full name of third joint inventor, if any (given name, family name) Barry William Luke Southward

Third inventor's signature [Signature]

Date

28/4/02

Residence ~~Belfast, Northern Ireland~~ 150 Cherry Sutton Road, Widnes, Cheshire WA8 4TJ, PWS

Citizenship British

United Kingdom. GBX

Post Office Address 19 Ardmore Avenue

Formerly of: Ormeau, Belfast BT7 3HD, Northern Ireland

4-0
Full name of fourth joint inventor, if any (given name, family name) David Walls

Fourth inventor's signature David Walls

Date

28.7.02.

Residence Derbyshire, United Kingdom

Citizenship British

Post Office Address 4 Whitecotes Park

Chesterfield, Derbyshire S40 3RT, United Kingdom

Full name of fifth joint inventor, if any (given name, family name) _____

Fifth inventor's signature _____

Date _____

Residence _____

Citizenship _____

Post Office Address _____

Full name of sixth joint inventor, if any (given name, family name) _____

Sixth inventor's signature _____

Date _____

Residence _____

Citizenship _____

Post Office Address _____

Full name of seventh joint inventor, if any (given name, family name) _____

Seventh inventor's signature _____

Date _____

Residence _____

Citizenship _____

Post Office Address _____